

ADSORPTION OF NATURAL GAS AND
HYDROGEN ON $\text{Cu}_3(\text{btc})_2$

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I declare that this thesis entitled Adsorption of Natural Gas and Hydrogen on $\text{Cu}_3(\text{btc})_2$ is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :

Name :

Date :

*Dedicated, in thankful appreciation for support,
encouragement and understanding
to my beloved family and friends.*

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In the name of Allah S.W.T. the most gracious and most merciful, Lord of the universe, with His permission Alhamdulillah the study has been completed. Praise to Prophet Muhammad S.A.W., His companions and to those on the path as what He preached upon, might Allah Almighty keep us His blessing and tenders.

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ABSTRACT

Metal organic framework (MOFs) is capable of storing large amount of gases. $\text{Cu}_3(\text{btc})_2$ is one of the most promising storage materials. The purpose of this research is to develop and characterize the adsorbent, $\text{Cu}_3(\text{btc})_2$ as a storage of the methane and hydrogen for NGV and fuel cell application at room temperature and at low pressure. The methane and hydrogen adsorption into the metal organic frameworks was examined with two different parameters; pressure and time of adsorption. For the methane, the pressure were used are 2.0 bars, 1.5 bars and 1.0 bars. Meanwhile, for the hydrogen, the pressure were used are 6.0 bars, 4 bars and 2.0 bars. The time of adsorption that were used for methane and hydrogen are 1 hour, 2 hours, 3 hours and 4 hours respectively. The result of the experiment showed that the weight of $\text{Cu}_3(\text{btc})_2$ sample after the adsorption with hydrogen was decreased. Or in other word, the $\text{Cu}_3(\text{btc})_2$ sample did not adsorb hydrogen or adsorbed the hydrogen in very small quantity. This is because hydrogen only adsorb on $\text{Cu}_3(\text{btc})_2$ at lower temperature which is 77K(-196°C). The second reason was the $\text{Cu}_3(\text{btc})_2$ sample was not crystallized as there was no proper autoclaves to synthesis sample at 393 K and 12 hour. For the methane adsorption on $\text{Cu}_3(\text{btc})_2$, the weight of the sample after adsorption was increased. Hence, this proved that $\text{Cu}_3(\text{btc})_2$ can absorb the methane. However, the percentage of increased weight of the sample was quite low. This was due to the pressure that were used for methane adsorption on $\text{Cu}_3(\text{btc})_2$ were very low which are from 1.0 bar to 2.0 bar only. The characteristic of $\text{Cu}_3(\text{btc})_2$ was examined using Scanning Electron Microscope (SEM) to observe the structure of the metal alloy before and after adsorption. From the observation, it is showed that the adsorption was occurred in the $\text{Cu}_3(\text{btc})_2$ as the sample structure was expanded and the small pore of the sample was decreased. As a conclusion, the pressure and temperature of adsorption, pore size and surface area of sample are the essential parameter for the adsorption.

ABSTRAK

“Metal Organic Frameworks (MOFs)” mampu menyimpan sejumlah gas yang besar. $\text{Cu}_3(\text{btc})_2$ adalah salah satu MOFs yang mampu menyimpan gas dengan baik. Tujuan penyelidikan ini adalah untuk membangunkan dan mengkaji sifat $\text{Cu}_3(\text{btc})_2$ sebagai storan kepada gas metana dan hydrogen bagi kegunaan “Natural Gas Vehicle (NGV)” dan juga “fuel cell” pada suhu bilik dan tekanan yang rendah. Penjerapan gas metana dan hydrogen ke dalam $\text{Cu}_3(\text{btc})_2$ dikaji dengan keadaan tekanan dan masa penjerapan yang berbeza. Bagi gas metana, tekanan yang digunakan adalah 2.0 bar, 1.5 bar dan 1.0 bar. Manakala bagi gas hydrogen, tekanan yang digunakan adalah 6.0 bar, 4.0 bar dan juga 2.0 bar. Masa penjerapan bagi kedua-dua gas ini adalah 1 jam, 2 jam, 3 jam dan 4 jam. Hasil keputusan eksperimen ini menunjukkan berat sampel selepas penjerapan adalah berkurangan berbanding berat asal. Ini menunjukkan bahawa tiada penjerapan berlaku ataupun hanya sedikit sahaja yang berlaku. Keadaan ini terjadi disebabkan oleh $\text{Cu}_3(\text{btc})_2$ hanya boleh menyerap hydrogen dengan berkesan pada suhu 77 K (-196°C). Sebab yang kedua pula adalah kerana sampel $\text{Cu}_3(\text{btc})_2$ tidak berada dalam keadaan kristal. Ini berikutan ketiadaan alat autoklaf bersesuaian di makmal ketika eksperimen ini dijalankan. Bagi penjerapan menggunakan gas metana, berat sampel didapati bertambah. Ini membuktikan bahawa $\text{Cu}_3(\text{btc})_2$ mampu menyerap gas metana. Namun penjerapan ini berlaku dalam kuantiti yang sedikit. Ini berikutan tekanan yang digunakan adalah rendah iaitu dari 1.0 bar ke 2.0 bar sahaja. Siri sampel $\text{Cu}_3(\text{btc})_2$ telah dikaji menggunakan “Scanning Electron Microscope (SEM)” untuk melihat struktur sampel sebelum dan selepas penyerapan berlaku. Hasil eksperimen menunjukkan penjerapan telah berlaku di dalam sampel $\text{Cu}_3(\text{btc})_2$ di mana struktur sampel telah memanjang dan bilangan liang yang kecil semakin berkurangan sebaik sahaja penjerapan berlaku. Sebagai kesimpulan, tekanan dan suhu penjerapan, saiz liang, dan luas permukaan sampel mempengaruhi penjerapan ke atas sampel.

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LIST OF SYMBOLS

| | | |
|---|---|-----------------------|
| C | - | Celsius |
| F | - | Fahrenheit |
| g | - | Gram |
| K | - | Kelvin |
| L | - | Liter |
| m | - | Mass of adsorbent |
| P | - | Pressure |
| x | - | Quantity of adsorbent |
| T | - | Temperature |
| % | - | Percent |

LIST OF ABBREVIATIONS

| | | |
|--------------------------------|---|--|
| ANG | | Adsorb Natural Gas |
| Atm | - | Atmosphere |
| CH ₄ | - | Methane |
| C ₂ H ₆ | - | Propane |
| C ₃ H ₈ | - | Propane |
| C ₄ H ₁₀ | - | Butane |
| CNG | - | Compressed Natural Gas |
| COFs | - | Covalent Organic Frameworks |
| H ₂ | - | Hydrogen |
| LNG | - | Liquefied Natural Gas |
| NG | - | Natural Gas |
| NGL | - | Natural Gas Liquid |
| NGV | - | Natural Gas Vehicle |
| MOFs | - | Metal Organic-Frameworks |
| ppm | - | Part per million |
| psi | - | Pound per square inch (lb/in ²) |
| psia | - | Pound per square inch of absolute pressure |
| psiag | - | Pound per square inch of gauge pressure |
| SEM | - | Scanning Electron Microscope |
| STP | - | Standard Temperature Pressure |
| v/v | - | Volume of adsorbed gas per volume of solid adsorbent |

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CHAPTER 1

INTRODUCTION

1.1 Overview

Gas storage is a container to holding liquid or vapour gas. It is defined as a gas storing vessel to accommodate fluctuating gas demands. Total gas demand usually varies considerably from summer to winter in western country. Usually it store liquid gas in high pressure condition and can be underground or aboveground. It can be used for both reservoir (large volume storage) and manufacture container. The manufacturer containers are available in many forms such as vertical and horizontal cylindrical.

They are two type of gas storage. First is underground storage which is used a sub-surface structure as a gas storage. Second is storage in vessels. First type of storage is divided by depleted gas reservoir, aquifer reservoir and salt formation.

Depleted gas reservoir is underground storage that can be used to store large volume of natural gas. This storage is made from the reservoir formation of natural gas field that have produced all their economically recoverable gas. This reservoir usually well known because their geological and physical characteristics have been already studied by geologists and petroleum engineers. Consequently, depleted reservoirs are generally the cheapest and easiest to develop, operate, and maintain of the three types of underground storage.

Aquifer reservoirs are underground, porous and permeable rock formation that act as natural gas reservoir. Usually these facilities are operated on a single annual cycle as with depleted reservoirs. The geological and physical characteristics of aquifer formation are not known ahead of time and a significant investment has to go into investigating these and evaluating the aquifer's suitability for natural gas storage.

Salt formation or salt cavern usually much smaller than depleted gas reservoir and aquifer storage facilities. A salt cavern facility may occupy only one one-hundredth of the area taken up by a depleted gas reservoir facility. Consequently, salt caverns cannot hold the large volumes of gas necessary to meet base load storage requirements. Deliverability from salt caverns is, however, much higher than for either aquifers or depleted reservoirs. This allows the gas stored in a salt cavern to be withdrawn and replenished more readily and quickly. This quick cycle-time is useful in emergency situations or during short periods of unexpected demand surges.

Although construction is more costly than depleted field conversions when measured on the basis of dollars per thousand cubic feet of working gas, the ability to perform several withdrawal and injection cycles each year reduces the effective cost.

Second type of storage is storage in vessel. It can be divided by low pressure holder and high pressure holder. Low pressure holder is a traditional form of gas storage. Usually it can be found in a former gas manufacturing plant. It designs to 'throw' a gas pressure of a few bar on the local distribution network by virtue of their own weight.

In the high pressure holder, a large quantity of gas can be stored under pressure in a comparatively small physical volume by gas compression. The gas volume is reduced by a factor equal to the ratio of the absolute pressure. The main types of high pressure holder are high pressure storage, pipe arrays and line pack.

Usually, methane and hydrogen are stored at lower temperature and high pressure. The conventional method to store hydrogen and methane is by cooling at low temperature or compress at high pressure. To make storage easy, effective, and safely, research are done to find storage at low pressure and at room temperature. The new technique to store this gas at low pressure and room temperature is gas adsorption. It means that gas was adsorbing into the surface of solid. The adsorbent usually used is activated carbon, metal hydride alloy, metal organic frameworks, covalent organic frameworks and others. (US Patent 6613126, 2003)

1.2 Identification of Problem

In a recent year, oil as a fossil fuel and a world source of energy has increased rapidly because of several factors. So the demand of using natural gas as a vehicle fuel is increased. There are driven by compressed natural gas (CNG) and few cases drive by liquefied natural gas. (LNG). However, to store natural gas using LNG is the most significant problems regard is that methane is in a supercritical state near room temperature. LNG offers an energy density comparable to diesel and fuel, but the method is expensive especially for small car. CNG has as a specific volumetric storage capacity about 240 v/v at 200 bar, which represents the volume of stored methane at ambient conditions (1 bar, 303 K) per unit volume of vessel. This is about 2.6 times lower than for natural gas in the liquid state. Therefore, alternative methods of methane storage have been intensively studied in recent years (Lee *et al.*, 2006).

Currently, as a promising method, the storage of methane as adsorbed gas in porous materials is discussed. The combination of CNG vessels adsorbing natural gas (ANG) as a storage medium could be competitive with current and LNG vehicles and would afford a higher gas storage density. Therefore, the development of new materials suitable for methane adsorption is an existing challenge and is an active area of research (Lee *et al.*, 2006).

The main challenges of ANG storage products development are:-

- i. To afford a higher gas storage capacity for the porous material
- ii. ANG fuelling system cost that should be competitive with the cost of existing fuelling system
- iii. Sufficient volumetric storage ability that will be competitive with existing NG storage methods.
- iv. Efficient gas filling and release from ANG tank for automotive application, requiring control of thermo-dynamic processes

Another way to replace oil as a main source of energy is a fuel cell. In addition to the fuel cell, the fuel cell vehicle carries a hydrogen storage tank which stores hydrogen gas to be supplied to the fuel cell. Hydrogen is a non-fossil fuel gas and also cleans gas because only produce only water which is quite harmless. In fuel cell, hydrogen store at high pressure. In the high pressure hydrogen storage tank, the larger the charging pressure is, the larger the storage amount of the hydrogen gas is. Accordingly, the amount of the hydrogen gas capable of being supplied to the fuel cell is increased due to the large charging pressure. Therefore it is necessary to increase the charging pressure to be as high as possible.

However, they have a limitation in container. So in a past research they use metal alloy as hydrogen storage. But hydrogen was chemically bound to the metal, not physically bound. That means we need to pressurize the material to charge it with hydrogen, and we have to heat the material to high temperatures to discharge the hydrogen. The process of charging and discharging under these extreme conditions ends up contaminating the metal and breaking the whole process down, so these materials have a limited lifetime.

1.3 Objective

The objective of this research is to develop and characterize the adsorbent, $\text{Cu}_3(\text{btc})_2$ as a storage of the methane and hydrogen for NGV and fuel cell application at room temperature and at low pressure.

1.4 Scope of Study

Scope of study in this research is to identify:-

- i. To identify the impact of using $\text{Cu}_3(\text{btc})_2$ as storage medium for H_2 and CH_4 .
- ii. The characterization $\text{Cu}_3(\text{btc})_2$ as adsorbent of H_2 and CH_4 .
- iii. To study the performance of $\text{Cu}_3(\text{btc})_2$ as adsorbent when changing pressure, and time adsorption.

1.5 Rational and Significant

The rational and the significant of this research is because of fuel price as a source of energy is increasing and we need to find other source of energy. Beside of that, we also need a clean fuel that produce less emission gas to protect our environmental. Natural gas and hydrogen are a most suitable source.

Natural gas (NG) has a considerable advantage over conventional fossil fuels both from an environmental point of view and due to the natural abundance and resources. The main component of natural gas is methane, with a high heat of combustion. The combustion of methane produces the smallest amount of carbon dioxide per unit of heat produced among fossil fuels. Thus a further improvement of

natural gas driven vehicles is a key issue for the development of environmentally friendly transportation systems.

The energy density per unit volume of hydrogen is significantly less than other fuel source but hydrogen only produce water as a combustion product. So it produces only Nox emission, not carbon emission. Hydrogen also is non-fossil fuel and was dubbed as a future source of energy.

Conventional energy storage is not fully safe and expensive. It exposed to explosion because it stored at high pressure and need expensive raw material to manufactured storage tank. To overcome this problem, a new material that can store a gaseous at low pressure and at room temperature must be developing. This material also must provide large storage capacity at low cost and have high efficiency of adsorption and desorption rate.

CHAPTER 2

LITERATURE REVIEW

2.1 Adsorption

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of solid, forming a film molecules or atoms. And absorption is a process when gas or liquid brought into contact with a solid and taken into the solid. Or in other word, atoms, molecules, or ions enter some bulk phase-gas, liquid or solid material. When both of this process takes place simultaneously, it's called sorption. And desorption is a process where a substance is released from or through a surface.

Solid can be a good adsorption if it has a surface composed of molecules which provide a good attractive force. When molecules in the gas or liquid pass near the surface of the solid, they stick. This can happen in one of two ways:

- i. Physisorption (by van der Waals and electrostatic forces)

This is the most common form of adsorption. The molecules are attracted by van der Waals forces, and attach themselves to the surface of the solid. The molecules remain intact, and can be freed easily (the forces are small, and short-range).

ii. Chemisorption (by chemical bonding)

The molecules undergo a chemical bonding with the molecules of the solid, and this attraction may be stronger than the forces holding the solid together. If the molecules are removed, they may form different compounds.

Other requirement to be a good adsorbent is a large surface area. The bigger its surface area, the more molecules it can trap on its surface. It means that good adsorbent will be very porous.

Adsorption is usually described through isotherms, the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials.

The first mathematical fit to an isotherm was published by Freundlich and Kuster (1894) and is a purely empirical formula for gaseous adsorbent's,

$$\frac{x}{m} = kP^{\frac{1}{n}}$$

where x is the quantity adsorbed, m is the mass of the adsorbent, P is the pressure of adsorbate and k and n are empirical constants for each adsorbent adsorbate pair at a given temperature. The function has an asymptotic maximum as pressure increases without bound. As the temperature increases, the constants k and n change to reflect the empirical observation that the quantity adsorbed rises more slowly and higher pressures are required to saturate the surface.

Others isotherm model is Langmuir equation publishes by Irving Langmuir (1916) and this equation was argue by BET theory publish by Stephan Brunauer, Paul Emmett, and Edward Teller (1938).

2.2 Natural Gas (NG)

Natural gas is colourless, highly flammable gaseous hydrocarbon consisting primarily of methane and ethane. It is a type of petroleum that commonly occurs in association with crude oil. Natural gas is often found dissolved in oil at the high pressures existing in a reservoir, and it also can be present as a gas cap above the oil. Such natural gas is known as associated gas. There are also reservoirs that contain gas and no oil. This gas is termed non-associated gas. The natural gas industry is producing gas from increasingly more challenging resource types: sour gas, tight gas, shale and coalbed methane.

The world's largest proven gas reserves are located in Russia, with $4.757 \times 10^{13} \text{ m}^3$ ($1.6 \times 10^{15} \text{ cu ft}$). Russia is also the world's largest natural gas producer, through the Gazprom company. Major proven resources (with year of estimate) (in billion cubic meters) are world 175,400 (2006), Russia 47,570 (2006), Iran 26,370 (2006), Qatar 25,790 (2007), Saudi Arabia 6,568 (2006) and United Arab Emirates 5,823 (2006). The world's largest gas field is Qatar's offshore North Field, estimated to have 25 trillion cubic meters ($9.0 \times 10^{14} \text{ cu ft}$) of gas in place, enough to last more than 200 years at optimum production levels. (<http://www.state.gov/r/pa/ei/bgn/5437.htm>, October 2008) The second largest natural gas field is the South Pars Gas Field in Iranian waters in the Persian Gulf. Connected to Qatar's North Field, it has estimated reserves of 8 to 14 trillion cubic meters (2.8×10^{14} to $5.0 \times 10^{14} \text{ cu ft}$) of gas.

Because natural gas is not a pure product, when non-associated gas is extracted from a field under supercritical (pressure/temperature) conditions, it may partially condense upon isothermic depressurizing--an effect called retrograde condensation. The liquids thus formed may get trapped by depositing in the pores of the gas reservoir. One method to deal with this problem is to re-inject dried gas free of condensate to maintain the underground pressure and to allow re-evaporation and extraction of condensates. Huge quantities of natural gas (primarily methane) exist in the form of hydrates under

sediment on offshore continental shelves and on land in arctic regions that experience permafrost such as those in Siberia (hydrates require a combination of high pressure and low temperature to form). However, as of 2008, there is no technology that has been developed to recover it economically.

The primary component of natural gas is methane (CH_4), the shortest and lightest hydrocarbon molecule. It often also contains heavier gaseous hydrocarbons such as ethane (C_2H_6), propane (C_3H_8) and butane (C_4H_{10}), as well as other sulphur. Natural gas that contains hydrocarbons other than methane is called wet natural gas. Natural gas consisting only of methane is called dry natural gas. Table 2.1 shows the typical composition of natural gas and LPG in Malaysia.

Table 2.1: Typical Malaysia natural gas and LPG composition

| Item | | Natural Gas (before 1995) | Natural Gas (after 1995) | LPG |
|----------------|------------------------------------|------------------------------|-----------------------------|-----|
| Gas Component | | | | |
| Methane | CH_4 (vol. %) | 84.75 | 92.74 | |
| Ethane | C_2H_6 (vol. %) | 10.41 | 4.07 | |
| Propane | C_3H_8 (vol. %) | 0.98 | 0.77 | 30 |
| Butane | C_4H_{10} (vol. %) | 0.11 | 0.14 | 70 |
| Nitrogen | N_2 (vol. %) | 0.39 | 0.45 | |
| Carbon Dioxide | CO_2 (vol. %) | 3.36 | 1.83 | |

The image below (Figure 2.1) is a schematic block flow diagram of a typical natural gas processing plant. It shows the various unit processes used to convert raw natural gas into sales gas pipelined to the end user markets. The block flow diagram also shows how processing of the raw natural gas yields byproduct sulfur, byproduct ethane, and natural gas liquids (NGL) propane, butanes and natural gasoline (denoted as pentanes + or C_5+).